

**REMARKS**

**Specification has been amended to eliminate stilted phrases and clarify meaning.**

Several paragraphs throughout the specification have been amended to eliminate the stilted phrases "electrical contact enhancement means" and "electrical isolation means" and to clarify meaning. These amendments to the specification constitute mere rephrasing which adds no new material, whereby they should be admitted. *In re Anderson*, 471 F.2d 1237, 176 USPQ 331 (CCPA 1973); MPEP § 2163.07, part I.

In several places, references to related Patent Application 09/855,293, now US Patent 6,589,405 have been amended accordingly.

**Claims 10-14 have been amended to properly specify function and clarify meaning.**

Original claims 10-14 were rejected under 35 USC § 112, second paragraph as lacking a statement of function as required by 35 USC § 112, sixth paragraph. These claims have been amended to provide clear statements of function, whereby the equivalents of the functional elements can now readily be identified with reference to the specification.

**The limitations of Claim 2 have been added to Claim 1.**

The limitations of Claim 2 have been added to Claim 1. Original Claim 2 has been withdrawn as being redundant to Currently Amended Claim 1, and references to Claim 2 in Claims 3, 9, 10 and 13 have been amended to refer to Currently Amended Claim 1.

Original Claim 8 has been withdrawn as no longer properly dependent on Currently Amended Claim 1.

**Examiner has failed to establish a prima facie case of obviousness.**

Claims 1-20 have been rejected under 35 USC 103(a) as being unpatentable over Coin et al (US Patent 5,783,050) in combination with Amamoto (US Patent 6,325,875). In fact, Examiner has for several reasons failed to establish a prima facie case of obviousness, whereby the claims as currently amended SHOULD BE ALLOWED.

Amamoto patent is not available as prior art

The instant application is a continuation-in-part of regular application No. 09/855,293, filed on May 14, 2001, now US Patent 6,589,405. All but two of the original claims in the instant application (9 and 18, which claim crimping the Ti fiber tow) are fully supported by the earlier application 09/855,293. The Amamoto patent issued December 4, 2001, and the corresponding application (09/758,343) was published (2001/0003627) June 14, 2001.

Therefore, the Amamoto patent and the corresponding US patent publication are prior art only in respect to claims 9 and 18.

Examiner may wish to cite the corresponding Japanese publication instead

There is a Japanese patent publication (H11-081050, A, 26.03.1999) which is related to the Japanese patent application (H09-251322) which the Amamoto patent claims benefit of, but this earlier Japanese publication was not cited by Examiner. A copy of the computer generated translation provided by the Japan Patent Office website is enclosed herewith.

Amamoto does not provide the suggestion to combine with Coin

On page 5 of the Office Action, Examiner states that Amamoto provided the suggestion to combine: "The prior art of Amamoto discloses that it is known to make a titanium fiber electrode having a high specific surface area and that this fiber can be used in bundle form and can include a catalyst material."

In fact, there is no such statement or suggestion in the Amamoto patent, nor in the earlier Japanese publication. In fact, the words "electrode", "anode" and "cathode" nowhere appear in either of these documents, and in regard to uses for the Ti-fiber bundle, Amamoto suggests: "Extrafine metal fibers having a diameter of about 5-30 $\mu$ m are used in various fields such as a material for a filter or a catalyst carrier, or as a filler for giving an electrical conductivity of a strength to plastics, cloth and the like." (1:18-21) Amamoto makes no mention or suggestion of using Ti-fiber to make an electrode, in the form of a bundle or otherwise.

Amamoto's suggestion to use Ti-fiber as a catalyst support also fails to provide a suggestion to use Ti-fiber in an electrode structure, because it arises in a distant and unrelated field of art. Indeed, the highly porous structures favored in relation to catalysts would make very poor electrode coatings that allow massive leakage of current through the coating, resulting in

passivation of the Ti-metal substrate and rapid electrode failure.

Coin teaches away from combination with Amamoto

In several places in 5,783,050 Coin explicitly refers to his electrodes as “fiber-free” (4:6, (5:32, 6:13, claim 1 at 20:65, and claim 29 at 22:50) and states that his electrode, comprising an expanded metallic mesh wrapped around a rigid core member, is superior to earlier prior art electrodes incorporating metallic fibers in the form of a felted mat or stitched fiber fill because difficulty of attaching the fiber to a current collector are thereby avoided (6:13-16). Thus, Coin teaches away from combination with Amamoto. It is improper to combine references where the references teach away from their combination. *In re Graselli*, 713 F.2d 731 at 743, 218 USPQ 769, 779 (Fed. Cir. 1983); MPEP § 2145, part X.D.2.

Prior art provides no reasonable expectation of success

Kaczur et al. 5,298,280 (disclosed by applicant) teaches spot welding titanium fiber to a titanium plate to make a large surface area electrode. (Kaczur uses cut pieces of Ti-fiber, rather than a bundle of fibers.) According to Kaczur, interconnecting the fibers by metallurgical sintering, and attaching the fibers to the substrate plate by spot welding are preferred methods to provide good electrical contact and conductivity (6:6-15).

Kaczur also argues that “woven structures made from multi-filament strands (or tow bundles)” of metallic fiber provide “inadequate performance” because “the porosity of these structures is non-uniform, such that the zones with highest surface area do not allow penetration of current through the electrolyte between closely spaced fibers” (2:45-51). Kaczur makes no mention of wound structures like those provided in the instant application.

Thus, Kaczur teaches away from using a bundle of metallic fibers to produce an electrode structure. As noted above, Coin also teaches away from using metallic fiber in electrode structures.

Based on the teachings of Kaczur and Coin, one skilled in the art of electrode production would be led to conclude that merely winding a bundle (or tow) of Ti-fiber around a Ti plate would fail to produce a usable electrode. Without a reasonable expectation of success in the combination, a prima facie case of obviousness is lacking. *In re Vaeck*, 947 F.2d 488, 20 USPQ2d 1438 (Fed. Cir. 1991); MPEP § 2142.

**Prior art would defeat prima facie case of obviousness (if one were made)**

The consensus teachings of the prior art would defeat the prima facie case of obviousness, if one were actually made.

The teachings of Kaczur and Coin recited above effectively constitute “accepted wisdom” in relation to using bundles of metallic fiber to construct an electrode. In the instant case, applicants have “proceeded contrary to accepted wisdom [,and] this is strong evidence of nonobviousness.” *In re Hedges*, 783 F.2d 1038 at 1041, 228 USPQ 685 (Fed. Cir. 1986) and earlier references cited therein [internal quotation marks deleted]; MPEP § 2145, part X.D.3.

**“Crimping” fiber distinct from “flattening” fiber taught by Amamoto**

On page 5 of the Office Action, Examiner wrote “The recitation of flattening in the prior art of Amamoto disclosing the increased surface area appears to meet the ‘crimping’ limitation set forth in [dependent claims 9 and 18].” This conclusion is in error. Amamoto describes actually deforming the cross-sectional shape of the Ti-fibers from a nearly circular form to a flattened form with substantially greater circumference by hot-rolling the precursor Ti/carbon steel composite structure from which the fiber is obtained.

The present applicants recite “crimping” the fiber tow to give it a wavy form along the length of the tow. Crimping involves bending the fibers, and has essentially no effect upon the cross-sectional shape of the individual fibers. Applicants nowhere suggest that crimping increases the surface area of the fibers; rather, it gives the tow a “fluffy” structure, allowing a more uniform, more easily coated electrode structure to be produced. The two crimping gears 59 and 59A in Figure 3 of the instant application are merely intermeshed, with no external compressive force applied. The compressive force applied to the fiber tow as it pulled through between the two crimping gears is just sufficient to bend the fibers, and is very much smaller than the force that would need to be applied to actually flatten the cross-section of the fibers, which would be similar to the yield strength of titanium; that is, on the order of 100,000 psi.

Thus, there is nothing in the prior art of record that anticipates “crimping” the fiber tow.

**Crimping the fiber solves a long recognized problem**

Kaczur (quoted above) noted that electrode structures made by weaving metallic fiber tow are characterized by an uneven density of fibers resulting in poor electrode performance. Crimping the fiber as recited in the instant application solves this long-recognized problem in a simple and efficient manner, which supports a finding of nonobviousness.

**Amamoto's method of roughening the surface of the fiber is not prior art**

In the Office Action, Examiner suggests that Amamoto's method of increasing the surface area of the Ti fiber by creating a roughened microscopic surface texture is prior art against the instant application. In fact, it is not. Applicants' electrode, made by winding a tow comprising many fine metallic fibers about a conductive substrate member, relies on the large specific surface area inherent to fine fibrous materials regardless of the microscopic surface texture of the fibers. Applicants in no way claim or rely on the microscopic surface texture of the fiber, or the cross-sectional form of the fiber, whatever these may be; therefore, Amamoto's teachings in regard to the microscopic surface texture of the fibers and their cross-sectional form do not constitute prior art against the instant invention.

**Conclusion**

For all of the above reasons, applicants submit that the specification and claims as amended are now in proper form, and that the claims all define patentably over the prior art. Therefore they submit that this application is now in condition for allowance, which action they respectfully solicit.

**Conditional Request for Constructive Assistance**

Applicants have amended the specification and claims of this application so that they are proper, definite, and define novel structure which is also nonobvious. If, for any reason this application is not believed to be in full condition for allowance, applicants respectfully request the constructive assistance and suggestions of Examiner pursuant to M.P.E.P. § 706.03(d) and § 707.07(j) in order that the undersigned can place this application in allowable condition as soon

as possible and without the need for further proceedings.

Very respectfully,



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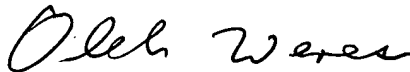
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<b>(54) TITANIUM FIBER AND ITS PRODUCTION</b>	
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PATENT ABSTRACTS OF JAPAN



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(54) TITANIUM FIBER AND ITS PRODUCTION

(57)Abstract:

PROBLEM TO BE SOLVED: To provide titanium fiber that has a larger specific surface area than the conventional titanium fiber of the same fiber diameter and a process that can steadily produce the objective fiber in high efficiency.

SOLUTION: In a metallic fiber 8 with circle-corresponding diameter  $d$  (in  $\mu\text{m}$ ) of  $5\ \mu\text{m}$  to  $30\ \mu\text{m}$  given by converging wire drawing, the material is titanium or its alloy and the metal fiber has a specific surface area  $A$  (in  $\text{m}^2/\text{g}$ ) satisfying  $A=25/d$ .

[Claim(s)]

[Claim 1] The metal fiber which construction material is [ the circle equivalent diameter  $d$  (micrometer) obtained by the focusing wire-drawing method ] titanium or a titanium alloy in a 5 to 30 micrometers metal fiber, and is characterized by specific surface area  $A$  ( $\text{m}^2/\text{g}$ ) satisfying  $A \geq 25/d$  (formula 1).

[Claim 2] The metal fiber given in the 1st term of a claim which specific surface area  $A$  ( $\text{m}^2/\text{g}$ ) does  $30/d \leq A \leq 50/d$  (formula 2) satisfaction of.

[Claim 3] The process which bundles several coat many strands 4 with which the enveloping layer 2 was formed in the perimeter of the metal strand 1, and is made with the bonnet compound wire rod 6 by the sheathing material 5, In the manufacture approach of the metal fiber which repeats and performs cold drawing processing and annealing heat treatment to this compound wire rod 6, removes the compound strand 7, the process to make, and the part equivalent to the enveloping layer 2 and sheathing material 5 in this compound strand 7, and includes the metal



fiber bundle 8 and the \*\*\*\* process to make The enveloping layer 2 of that they are a titanium wire rod or a titanium-alloy wire rod and the (b) coat strand 4 and the sheathing material 5 of a compound wire rod the (b) metal strand 1 The manufacture approach of the titanium fiber given in the 1st term of a claim characterized by making into 580 to 650 degrees C the highest attainment temperature of the compound wire rod in that it is the mild steel containing 0.25 or less % of the weight of carbon, and annealing heat treatment performed to the compound (Ha) wire rod 6, or titanium-alloy fiber.

[Claim 4] The process which manufactures the coat strand 4 is the manufacture approach of the titanium fiber given in the 3rd term of a claim which makes the highest attainment temperature of the covered-wire material 3 in annealing heat treatment 580 to 650 degrees C, or titanium-alloy fiber including the process which forms an enveloping layer 2 in the perimeter of the metal wire rod 1, and is made into the covered-wire material 3, and the process which gives at least one annealing heat treatment and cold-working \*\*\*\* to this covered-wire material 3.

[Claim 5] Thickness of the enveloping layer 2 of the covered-wire material 4 is made into 5 to 20% of the diameter of the coat strand 4. Total amount of processings  $\epsilon_T$  of the cold drawing given to the compound wire rod 6  $\{\epsilon_T = 2 \times \ln(DS/DF)\}$  (formula 3), DS The diameter of the compound wire rod 6 before performing cold drawing processing, and DF The diameter of the compound wire rod 6 after performing cold drawing processing, of the compound strand 7 to 5.5 to 7.5, titanium fiber given in the 4th term, or titanium-alloy fiber.

## DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Field of the Invention] As for this invention, a circle equivalent diameter is especially related with the large titanium fiber or the titanium-alloy fiber, and its manufacture approach of specific surface area about 5 to 30 micrometers titanium fiber or titanium-alloy fiber, and its manufacture approach. (In this invention, titanium and a titanium alloy are hereafter named "titanium" generically.)

[0002]

[Description of the Prior Art] The diameter is used as a filler for a 5 to about 30 micrometers super-thin metal fiber to give conductivity and reinforcement to a filter, the raw material of catalyst support or plastics, cloth, etc. As this kind of a super-thin metal fiber, the stainless steel fiber manufactured by the focusing wire-drawing method is used widely. On the other hand, it is more nearly lightweight than a corrosion resistance high filter, the raw material of catalyst support, or stainless steel fiber, the filler with high specific strength is called for rather than stainless steel fiber, and titanium fiber attracts attention. In the field of the catalyst whose surface area especially kept for a reaction is the important factor of the engine performance, titanium fiber with a more large specific surface area is called for.

[0003] As the manufacture approach of a 5 to about 30 micrometers super-thin metal fiber, the focusing wire-drawing method is learned for the diameter, for example, the following manufacture approaches are indicated. The metal fiber is laid underground into the copper matrix by JP,2-52117,A, the focusing wire-drawing complex (compound strand) which it comes to cover with a cylinder-like steel strip further is created to it, and the method of the permutation dissolution and electrolysis removing a copper matrix (enveloping layer) and a cylindrical steel strip (sheathing material), and acquiring a metal fiber bundle is indicated. However, since the

titanium manufactured by this manufacture approach has little surface irregularity, in order to obtain titanium fiber with a large specific surface area, it must make the diameter of fiber thin, and has the trouble that the time and effort and cost which processing takes increase.

[0004] Moreover, cold drawing is given to the covered-wire material which covered the wire rod made from a high anticorrosion alloy by the enveloping layer which a carbon content becomes from 0.12 or less % of the weight of steel, a coat strand is formed in JP,5-177244,A, these a majority of coat strands are inserted in a book into a steel pipe in a bundle, and a compound wire rod, nothing, and the method of giving cold drawing succeedingly, forming a compound strand, carrying out dissolution clearance of the part which is equivalent to a steel pipe and an enveloping layer with electrolysis, and acquiring a metal fiber bundle are indicated. However, although surface irregularity becomes large rather than what was manufactured by the approach indicated by above-mentioned JP,2-52117,A when this manufacture approach is applied to manufacture of titanium fiber, dissolution clearance in a final process is not fully performed, but there is a trouble that the yield is low.

[0005]

[Problem(s) to be Solved by the Invention] The object of this invention is to offer titanium fiber with a larger specific surface area than the conventional titanium fiber of the same diameter of fiber, and the method of manufacturing it certainly at high effectiveness based on the trouble of the above-mentioned conventional technique.

[0006]

[Means for Solving the Problem] Start the structure of the 1st metal fiber of this invention, and the circle equivalent diameter  $d$  (micrometer) obtained by the focusing wire-drawing method sets to a 5 to 30 micrometers metal fiber. Construction material is titanium or a titanium alloy, and specific surface area  $A$  ( $\text{m}^2 / \text{g}$ ) is preferably applied to  $A \geq 25/d$  (formula 1) and the metal fiber with which specific surface area  $A$  ( $\text{m}^2 / \text{g}$ ) is satisfied of  $30/d \leq A \leq 50/d$  (formula 2).

[0007] And the process which the 2nd of this invention bundles several coat many strands 4 with which the enveloping layer 2 was formed in the perimeter of the metal strand 1, and is made with the bonnet compound wire rod 6 by the sheathing material 5, In the manufacture approach of the metal fiber which repeats and performs cold drawing processing and annealing heat treatment to this compound wire rod 6, removes the compound strand 7, the process to make, and the part equivalent to the enveloping layer 2 and sheathing material 5 in this compound strand 7, and includes the metal fiber bundle 8 and the \*\*\*\* process to make The enveloping layer 2 of that they are a titanium wire rod or a titanium-alloy wire rod and the (b) coat strand 4 and the sheathing material 5 of a compound wire rod the (b) metal strand 1 The manufacture approach of the titanium fiber of said 1st invention characterized by making into 580 to 650 degrees C the highest attainment temperature of the compound wire rod in that it is the mild steel containing 0.25 or less % of the weight of carbon and annealing heat treatment performed to the compound (Ha) wire rod 6 or titanium-alloy fiber is started.

[0008] The process which manufactures the aforementioned coat strand 4 makes the highest attainment temperature of the covered-wire material 3 in annealing heat treatment 580 to 650 degrees C including the process which forms an enveloping layer 2 in the perimeter of the metal wire rod 1, and is made into the covered-wire material 3, and the process which performs at least one annealing heat treatment and cold working to this covered-wire material 3. Furthermore, what is necessary is just to make thickness of the enveloping layer 2 of the covered-wire material 4 into 5 to 20% of the diameter of the coat strand 4. And it is total amount of processings

epsilonT of the cold drawing given to the compound wire rod 6 still more preferably. The diameter of the compound wire rod 6 before {epsilonT = 2x1n (DS/DF) (formula 3) and DS perform cold drawing processing, and DF are the manufacture approaches of the titanium fiber which sets diameter} of the compound strand 7 to 5.5 to 7.5, or titanium-alloy fiber.

[0009]

[Embodiment of the Invention] The circle equivalent diameter d (micrometer) obtained by the focusing core wire method is 5 micrometers to 30 micrometers, and the titanium fiber which is the 1st of this invention has a large specific surface area A (m<sup>2</sup> / g), and specific surface area A (m<sup>2</sup> / g) satisfies  $30/d \leq A \leq 50/d$  (formula 2) for  $A \geq 25/d$  (formula 1) preferably. Here, it is a diameter of circle with the cross section of fiber, and the same area with the circle equivalent diameter d.

[0010] The semantics of definition by the formula 1 in this invention is the high specific surface area which is not obtained by the conventional manufacture approach, and it is stainless steel fiber comparison of the equivalent size currently generally used, this is equivalent to a twice [ about / more than ] as many specific surface area as this, and when it is used for catalyst support, a gas adsorption object, etc., it can expect remarkable lightweight-ization.

[0011] It is the field out of which a big difference comes even if it compares with the titanium fiber of the equivalent size by the conventional method about a minimum as a reason for definition of the desirable range. On the other hand, moreover, about an upper limit It specifies from a viewpoint of the ease of manufacture, and by size comparison of fiber, I hear that the surface irregularity of specific surface area being large is remarkable, and there is. When this upper limit is exceeded, the irregularity of the metal fiber adjoined in a compound wire rod becomes entangled mechanically, and there is a fault from which separation becomes difficult at a \*\*\*\* process.

[0012] In order to enlarge specific surface area A so that it is in a metal fiber and a formula 1 is satisfied, it is required to form minute irregularity in a front face and to earn surface area. Moreover, a desirable gestalt is as follows, although surface area can be earned even if it makes the configuration of a fiber cross section flat or incurvates it. That is, the outline configuration of a fiber cross section is made into configurations, such as an approximate circle form, an abbreviation ellipse form, and a rough convex polygon, and surface area is earned by forming much minute irregularity in a front face. Specific surface area can be enlarged without spoiling the workability and reinforcement when processing it into yarn, textile fabrics, the felt, etc. by doing in this way.

[0013] Drawing 2 is the mimetic diagram showing the example of the cross-section configuration of the titanium fiber of this invention. The outline configuration of the cross section of the titanium fiber 8 shown in this drawing 2 is an ellipse form, and has much minute irregularity on the front face. In addition, it can ask for specific surface area also including irregularity with a minute front face by measuring gas adsorption surface area with a BET adsorption method.

[0014] As construction material of the titanium fiber of this invention, pure titanium as shown, for example in a table 1, alpha alloy, an alpha-beta alloy, and beta alloy can be used. In addition, a table 1 shows the standard annealing heat treatment conditions over titanium, and is an extract from "the processing technique of titanium" for Japanese titanium associations.

[0015]

[A table 1]

[0016] Next, it explains, referring to drawing 1 about the gestalt of implementation of the manufacture approach of the titanium fiber which is the 2nd of this invention. The manufacture approach of the titanium fiber of this invention is a thing about the approach of manufacturing titanium fiber by the focusing core wire method. The process which bundles several coat many strands 4 with which the enveloping layer 2 was formed in the perimeter of the metal strand 1, and is made with the bonnet compound wire rod 6 by the sheathing material 5, Cold drawing processing and the above mentioned annealing heat treatment are repeated and performed to this compound wire rod 6, the compound strand 7, the process to make, and the part equivalent to the enveloping layer 2 and sheathing material 5 in this compound strand 7 are removed, and it has the following description in the manufacture approach of a metal fiber including the metal fiber bundle 8 and the \*\*\*\* process to make.

[0017] (b) The metal strand 1 should be titanium fiber or a titanium-alloy wire rod. For example, pure titanium as shown in the aforementioned table 1, alpha alloy, an alpha-beta alloy, and beta alloy are used.

[0018] (b) The enveloping layer 2 of the coat strand 4 and the sheathing material 5 of the compound wire rod 6 should be the mild steel containing 0.12 or less % of the weight of carbon. If it says especially, the construction material of the enveloping layer 2 of the coat strand 4 will be important, it will be desirable to make the sheathing material 5 of the compound wire rod 6 into the same construction material as the enveloping layer 2 of the coat strand 4, and this will be for making easy setting out of the annealing heat treatment conditions mentioned later.

[0019] One of the reasons which specifies especially the construction material of this enveloping layer 2 is for forming much irregularity in the front face of the titanium fiber 8 manufactured, and earning specific surface area. That is, mild steel is a polycrystal ingredient which has the crystal structure of a body-centered cubic lattice, and each crystal grain has the strong anisotropy to deformation. For this reason, if wire drawing of the compound wire rod 6 which bundled several coat many strands 4 which made mild steel the enveloping layer 2 and made titanium the core material 1, and was covered by the sheathing material 5 is carried out, as typically shown in drawing 4, each crystal grain of the mild steel which forms the enveloping layer 2 will carry out bow deformation in the cross section, and much irregularity will be formed in the front face of the titanium core material 1. For this reason, the specific surface area of the titanium fiber 8 removed and obtained will increase the part equivalent to an enveloping layer 2 and a sheathing material 5.

[0020] Since the crystal grain of an enveloping layer 2 deforms almost isotropic in wire drawing on the other hand when crystal ingredients, such as copper with the crystal structure of a body-centered cubic lattice, are made into an enveloping layer 2, when mild steel is made into an enveloping layer 2, the irregularity of a like is not obtained, but becomes the cross section as typically shown in drawing 3, and cannot be said to be a desirable gestalt.

[0021] In the manufacture approach of the titanium fiber of this invention, since specifying 0.25 or less % of the weight of the mild steel containing 0.12 or less % of the weight of carbon preferably as construction material of an enveloping layer 2 also especially in the polycrystal ingredient which has the crystal structure of a body-centered cubic lattice has low ingredient cost, workability is good and is because formation of the coat strand 4 is easy. If it is in carbon steel with which especially a carbon content exceeds 0.25 % of the weight, it is large, and it is in the middle of processing, and it is not [ whenever / by wire drawing / hardening / needs to increase the count of heat treatment, and ] desirable. Moreover, in heat treatment which made the highest

attainment temperature 580-650 degrees C, it becomes difficult to fully recover wire drawing nature. 0.25 or less % of the weight, with the above-mentioned technical problem being solvable, especially the thing that contains 0.12 or less % of the weight of carbon if it says is excellent in bending or weldability, and it becomes easy to form [ of an enveloping layer ] it.

[0022] Moreover, formation of the coat strand 4 can be easily carried out by covering the perimeter of the titanium wire rod 1 with sheet metal, such as SPCC and SPCE. Moreover, in order to make deep the depth of the surface irregularity of the titanium fiber 8 manufactured and to obtain titanium fiber with a more large specific surface area, it is desirable to thicken thickness of the mild steel enveloping layer 2 relatively to the diameter of the coat strand 4, but if it is made not much thick too much, it will become easy to produce the problem that the time amount which the \*\*\*\* process which removes the part equivalent to an enveloping layer 2 and a sheathing material 5 takes becomes long. Then, the range where the thickness of an enveloping layer 2 is desirable is 5% to 20% of the diameter of the coat strand 4, and the still more desirable range is 8% to 15%.

[0023] And although whenever [ bow / of each crystal grain of mild steel ] becomes large and titanium fiber with a large specific surface area is obtained so that the amount of cold drawing processings given to the compound strand material 6 which bundled several coat many strands 4 and was covered by the sheathing material 5 is enlarged, it becomes easy to produce the problem that the time amount which a \*\*\*\* process takes too becomes long. Then, total amount of processings  $\epsilon T$  of the cold drawing given to a compound wire rod (it is desirable to set  $\epsilon T = 2 \times \ln(DS/DF)$  (formula 3) to 5.5 to 7.5.) It is DS here. The diameter of the compound wire rod 6 before performing cold drawing processing, and DF It is the diameter of the compound strand 7.  $\epsilon T$  Since whenever [ bow / of the crystal grain of mild steel ] is small when it is 5.5 or less, the irregularity of titanium fiber is small, and specific surface area becomes so large and is not desirable, either. Moreover, it becomes [ in the case of 7.5 or more the surface irregularity of titanium fiber becomes intense, and / that the irregularity of the metal fiber adjoined in a compound wire rod becomes entangled mechanically, and they dissociate at a \*\*\*\* process ] difficult and is not desirable.

[0024] (c) Make into 580 to 650 degrees C the highest attainment temperature of the compound wire rod in annealing heat treatment performed to the compound wire rod 6. This temperature requirement is a temperature requirement where this invention persons set up an experiment and examination in piles about the annealing heat treatment conditions of giving many coat strands 4 with which the mild steel enveloping layer 2 was formed in the perimeter of the titanium strand 1 to the compound wire rod 6 further covered by the mild steel sheathing material 5 in \*\*\*\*.

[0025] The standard heat treatment conditions over the titanium which does not have a mild steel coat were understood that it is required to take into consideration the diffusion phenomenon in the interface of titanium and mild steel with the softening degree of the compound wire rod 6 in annealing heat treatment of the compound wire rod 6 containing the coat strand 4 with which the mild steel enveloping layer 2 was formed in the perimeter of the titanium strand 1 the place which is a thing as shown in the above mentioned table 1. Namely, if the highest attainment temperature exceeds 650 degrees C, since the alloy layer formed of diffusion of the interface of titanium and mild steel will progress, When an enveloping layer 2 tends to be removed and it is going to obtain titanium fiber 8, even if clearance of an enveloping layer 2 becomes difficult and titanium fiber 8 is obtained When the highest attainment temperature is less than 580 degrees C, on the other hand in core wire processing between the colds which the softening degree of the

compound wire rod 6 serves as imperfection, and continues, it becomes easy only for a part of titanium fiber in the compound strand 7 to be obtained, but for the yield to fall remarkably, and to produce an open circuit remarkably.

[0026] Thus, although it is required to make into 580 to 650 degrees C the highest attainment temperature in annealing heat treatment performed to the compound wire rod 6 at least in the manufacture approach of the titanium fiber of this invention When the process which forms the coat strand 4 before forming the compound wire rod 6 includes annealing heat treatment to the covered-wire material 3 which formed the mild steel enveloping layer 2 in the titanium core material 1, it is desirable to also make into the range of 580 to 650 degrees C the highest attainment temperature in annealing heat treatment performed to the covered-wire material 3. In addition, since titanium [ activity / front face ] is covered with mild steel when performing annealing heat treatment to the compound wire rod 6 or the covered-wire material 4, it can operate also by the furnace atmosphere applied to steel-wire material using a gas fired furnace, an electric furnace, etc.

[0027] In addition, in the manufacture approach of the titanium fiber of this invention, especially wire drawing performed to the compound wire rod 6 is considered as cold drawing processing for an alloy layer tending to develop into the interface of titanium and mild steel, in order to ease the anisotropy over processing and for the effectiveness of surface irregularity formation for titanium fiber to fall, if hot working in an elevated temperature is applied. As the approach of cold drawing processing, the dry wire drawing method by the hole dice, a wet drawing method, or a roller die is applicable. Moreover, since it is covered with mild steel, wire drawing of the front face of the compound wire rod 6 or the covered-wire material 4 can be carried out using the lubricant for wire drawing of steel wire.

[0028]

[Example] It explains to a detail further with the desirable example of this invention. The description of the titanium fiber 8 manufactured on five kinds of manufacture conditions shown in a table 2 through the yield and \*\*\*\* process of an attempt, the stability of a process, and the titanium fiber in a \*\*\*\* process in manufacture of the compound strand 7 which included much titanium fiber was compared.

[0029]

[A table 2]

[0030] In a table 2, an example 1 and an example 2 are examples according to the suitable conditions of the manufacture approach of the titanium fiber of this invention, and have set up more thickly than an example 1 the enveloping layer thickness of the covered-wire material 3 in an example 2. In addition, in order to make equivalent to an example 1 the circle equivalent diameter of the titanium fiber 8 manufactured in an example 2, it is the example which has set up the diameter of the final compound strand 7 thickly a little rather than the example 1.

[0031] The example 1 of a comparison is an example which set up lower than the range according to this invention the highest attainment temperature in annealing heat treatment of the compound wire rod 6. Moreover, the example 2 of a comparison is an example which set more highly than the range according to this invention the highest attainment temperature in annealing heat treatment of the compound wire rod 6 as reverse in the example 1 of a comparison. The example 3 of a comparison is an example which used copper as an enveloping layer 2. In

addition, since it is hard to diffuse copper in titanium compared with iron, it gave priority to the softening degree of the titanium by heat treatment in the example 3 of a comparison, and has set up the highest attainment temperature more highly than other examples.

[0032] In formation of an enveloping layer 2, the pure titanium core material 1 was inserted in the interior, forming a welded tube with a diameter of about 6mm with the strip made into an enveloping layer 2, wire drawing was carried out to the diameter of 4.3mm, and the wall and core material front face of tubing were stuck. The strip made into an enveloping layer 2 used the copper strip for the example 1 and the example 2, and the list in the example 3 of a comparison using the strip of SPCC in the example 1 of a comparison, and the example 2 of a comparison. Moreover, in formation of the compound wire rod 6, the bundle of the coat strand 4 was inserted in the interior, forming a welded tube with a diameter of about 6mm with the strip of SPCC, and wire drawing was carried out to the diameter of 4.3mm, and it screwed up.

[0033] Heat treatment was performed continuously through the wire rod into the electric furnace set as predetermined temperature, except having made the heat treatment ambient atmosphere of the covered-wire material 3 in the example 3 of a comparison into the inert atmosphere, it was heat-treated by the weak oxidizing atmosphere, and the wire drawing which continues after making a front face into clarification by acid washing and rinsing was presented with it. Wire drawing applied the cold drawing by dry type and wet, and performed it using the lubricant for steel wire.

[0034] the result of having tried manufacture of the compound strand 7 which included much titanium fiber 8 on five kinds of manufacture conditions shown in a table 2 -- the heat treatment temperature of the compound wire rod 6 -- the likeness of excess -- in the example 1 of a comparison set up low, although the compound strand 7 of the diameter which open circuits occur frequently in the last wet drawing process, and is made into the object was not obtained. The compound strand 7 of the diameter made into the object in other examples was able to be obtained.

[0035] In a concrete production process, if a carbon content and workability are described, and the charge of carbon content uses the carbon steel which is about 0.55 % of the weight, for example, the part welded at the time of welded tube formation will break at the time of wire drawing, and will not bear processing on the way. Moreover,  $\epsilon_T$  If attached, if the example 2 was in the example of 6.14 and others, it was 6.35, and all could carry out wire drawing and \*\*\*\* processing satisfactory, and the titanium fiber of the specific surface area made into the object was obtained.

[0036] Then, \*\*\*\* processing was performed to the compound strand 7 manufactured at four kinds of processes except the example 1 of a comparison, and the description of the titanium fiber 8 obtained with the yield of an attempt and a \*\*\*\* process in manufacture of the titanium fiber bundle 8 was compared. The result is shown in a table 3. In addition, \*\*\*\* processing was performed by electrolyzing selectively the part equivalent to the sheathing material 5 and enveloping layer 2 in the compound strand 7 in the electrolytic solution containing a sulfuric acid.

[0037] The yield of a \*\*\*\* process is the rate separated as titanium fiber 8 among the titanium wire rods which the longest also made 1 hour time amount of electrolysis at a \*\*\*\* process, and were laid underground into the compound strand 7. Moreover, measurement of the specific surface area of titanium fiber 8 measured the nitrogen amount of adsorption with the BET adsorption method, and it was converted into the surface area per unit quantity, and it evaluated

it.

[0038]

[A table 3]

[0039] As shown in a table 3, even if it covered the compound strand 7 of the example 2 of a comparison which set up highly the heat treatment temperature of the compound wire rod 6 too much over electrolysis time amount in the \*\*\*\* process for 1 hour, it could not be \*\*\*\*(ed) thoroughly, but its yield was remarkably low. On the other hand, the compound wire rod 7 of the example 1 and example 2 according to the manufacture approach of this invention was able to be thoroughly \*\*\*\*(ed) by electrolysis of less than 1 hour. The cross section of the obtained titanium fiber 8 is a configuration as shown in drawing 2, and much irregularity was formed in the front face, and specific surface area was so large that it satisfied the formula 1, and was able to adsorb a lot of nitrogen gas per unit weight. Moreover, in the example 2 set up more thickly than the thickness of an enveloping layer 2, titanium fiber with a larger specific surface area than an example 1 was able to be obtained.

[0040] Moreover, although the compound strand 7 of the example 3 of a comparison which used construction material of an enveloping layer as copper was able to be \*\*\*\*(ed) thoroughly, its front face of the obtained titanium fiber 8 was smoother than the front face of the titanium fiber chosen from the compound wire rod 7 of an example 1 and an example 2. And the specific surface area was smaller than the titanium fiber obtained from the compound wire rod of an example 1 and an example 2, and was not what satisfies a formula 1.

[0041]

[Effect of the Invention] As explained above, the titanium fiber of this invention has a larger specific surface area than the conventional titanium fiber obtained by the focusing wire-drawing method. For this reason, when this is used as ingredients, such as a catalyst, catalyst support, or a gas adsorption object, it is more nearly lightweight than before and what has the high engine performance can be obtained. Moreover, by the manufacture approach of the titanium fiber of this invention, it is stabilized and titanium fiber with a large specific surface area can be manufactured by the high yield.

[Field of the Invention] As for this invention, a circle equivalent diameter is especially related with the large titanium fiber or the titanium-alloy fiber, and its manufacture approach of specific surface area about 5 to 30 micrometers titanium fiber or titanium-alloy fiber, and its manufacture approach. (In this invention, titanium and a titanium alloy are hereafter named "titanium" generically.)

## PRIOR ART

[Description of the Prior Art] The diameter is used as a filler for a 5 to about 30 micrometers super-thin metal fiber to give conductivity and reinforcement to a filter, the raw material of catalyst support or plastics, cloth, etc. As this kind of a super-thin metal fiber, the stainless steel fiber manufactured by the focusing wire-drawing method is used widely. On the other hand, it is more nearly lightweight than a corrosion resistance high filter, the raw material of catalyst support, or stainless steel fiber, the filler with high specific strength is called for rather than



stainless steel fiber, and titanium fiber attracts attention. In the field of the catalyst whose surface area especially kept for a reaction is the important factor of the engine performance, titanium fiber with a more large specific surface area is called for.

[0003] As the manufacture approach of a 5 to about 30 micrometers super-thin metal fiber, the focusing wire-drawing method is learned for the diameter, for example, the following manufacture approaches are indicated. The metal fiber is laid underground into the copper matrix by JP,2-52117,A, the focusing wire-drawing complex (compound strand) which it comes to cover with a cylinder-like steel strip further is created to it, and the method of the permutation dissolution and electrolysis removing a copper matrix (enveloping layer) and a cylindrical steel strip (sheathing material), and acquiring a metal fiber bundle is indicated. However, since the titanium manufactured by this manufacture approach has little surface irregularity, in order to obtain titanium fiber with a large specific surface area, it must make the diameter of fiber thin, and has the trouble that the time and effort and cost which processing takes increase.

[0004] Moreover, cold drawing is given to the covered-wire material which covered the wire rod made from a high anticorrosion alloy by the enveloping layer which a carbon content becomes from 0.12 or less % of the weight of steel, a coat strand is formed in JP,5-177244,A, these a majority of coat strands are inserted in a book into a steel pipe in a bundle, and a compound wire rod, nothing, and the method of giving cold drawing succeedingly, forming a compound strand, carrying out dissolution clearance of the part which is equivalent to a steel pipe and an enveloping layer with electrolysis, and acquiring a metal fiber bundle are indicated. However, although surface irregularity becomes large rather than what was manufactured by the approach indicated by above-mentioned JP,2-52117,A when this manufacture approach is applied to manufacture of titanium fiber, dissolution clearance in a final process is not fully performed, but there is a trouble that the yield is low.

## EFFECT OF THE INVENTION

[Effect of the Invention] As explained above, the titanium fiber of this invention has a larger specific surface area than the conventional titanium fiber obtained by the focusing wire-drawing method. For this reason, when this is used as ingredients, such as a catalyst, catalyst support, or a gas adsorption object, it is more nearly lightweight than before and what has the high engine performance can be obtained. Moreover, by the manufacture approach of the titanium fiber of this invention, it is stabilized and titanium fiber with a large specific surface area can be manufactured by the high yield.

## TECHNICAL PROBLEM

[Problem(s) to be Solved by the Invention] The object of this invention is to offer titanium fiber with a larger specific surface area than the conventional titanium fiber of the same diameter of fiber, and the method of manufacturing it certainly at high effectiveness based on the trouble of the above-mentioned conventional technique.

## MEANS

[Means for Solving the Problem] Start the structure of the 1st metal fiber of this invention, and the circle equivalent diameter  $d$  (micrometer) obtained by the focusing wire-drawing method sets to a 5 to 30 micrometers metal fiber. Construction material is titanium or a titanium alloy, and specific surface area  $A$  ( $m^2 / g$ ) is preferably applied to  $A \geq 25/d$  (formula 1) and the metal fiber

with which specific surface area  $A$  ( $\text{m}^2 / \text{g}$ ) is satisfied of  $30/d \leq A \leq 50/d$  (formula 2).

[0007] And the process which the 2nd of this invention bundles several coat many strands 4 with which the enveloping layer 2 was formed in the perimeter of the metal strand 1, and is made with the bonnet compound wire rod 6 by the sheathing material 5, In the manufacture approach of the metal fiber which repeats and performs cold drawing processing and annealing heat treatment to this compound wire rod 6, removes the compound strand 7, the process to make, and the part equivalent to the enveloping layer 2 and sheathing material 5 in this compound strand 7, and includes the metal fiber bundle 8 and the \*\*\*\* process to make The enveloping layer 2 of that they are a titanium wire rod or a titanium-alloy wire rod and the (b) coat strand 4 and the sheathing material 5 of a compound wire rod the (b) metal strand 1 The manufacture approach of the titanium fiber of said 1st invention characterized by making into 580 to 650 degrees C the highest attainment temperature of the compound wire rod in that it is the mild steel containing 0.25 or less % of the weight of carbon and annealing heat treatment performed to the compound (Ha) wire rod 6 or titanium-alloy fiber is started.

[0008] The process which manufactures the aforementioned coat strand 4 makes the highest attainment temperature of the covered-wire material 3 in annealing heat treatment 580 to 650 degrees C including the process which forms an enveloping layer 2 in the perimeter of the metal wire rod 1, and is made into the covered-wire material 3, and the process which performs at least one annealing heat treatment and cold working to this covered-wire material 3. Furthermore, what is necessary is just to make thickness of the enveloping layer 2 of the covered-wire material 4 into 5 to 20% of the diameter of the coat strand 4. And it is total amount of processings  $\epsilon_T$  of the cold drawing given to the compound wire rod 6 still more preferably. The diameter of the compound wire rod 6 before  $\{\epsilon_T = 2 \times \ln(DS/DF)\}$  (formula 3) and  $DS$  perform cold drawing processing, and  $DF$  are the manufacture approaches of the titanium fiber which sets diameter} of the compound strand 7 to 5.5 to 7.5, or titanium-alloy fiber.

[0009]

[Embodiment of the Invention] The circle equivalent diameter  $d$  (micrometer) obtained by the focusing core wire method is 5 micrometers to 30 micrometers, and the titanium fiber which is the 1st of this invention has a large specific surface area  $A$  ( $\text{m}^2 / \text{g}$ ), and specific surface area  $A$  ( $\text{m}^2 / \text{g}$ ) satisfies  $30/d \leq A \leq 50/d$  (formula 2) for  $A \geq 25/d$  (formula 1) preferably. Here, it is a diameter of circle with the cross section of fiber, and the same area with the circle equivalent diameter  $d$ .

[0010] The semantics of definition by the formula 1 in this invention is the high specific surface area which is not obtained by the conventional manufacture approach, and it is stainless steel fiber comparison of the equivalent size currently generally used, this is equivalent to a twice [ about / more than ] as many specific surface area as this, and when it is used for catalyst support, a gas adsorption object, etc., it can expect remarkable lightweight-ization.

[0011] It is the field out of which a big difference comes even if it compares with the titanium fiber of the equivalent size by the conventional method about a minimum as a reason for definition of the desirable range. On the other hand, moreover, about an upper limit It specifies from a viewpoint of the ease of manufacture, and by size comparison of fiber, I hear that the surface irregularity of specific surface area being large is remarkable, and there is. When this upper limit is exceeded, the irregularity of the metal fiber adjoined in a compound wire rod becomes entangled mechanically, and there is a fault from which separation becomes difficult at a \*\*\*\* process.

[0012] In order to enlarge specific surface area A so that it is in a metal fiber and a formula 1 is satisfied, it is required to form minute irregularity in a front face and to earn surface area. Moreover, a desirable gestalt is as follows, although surface area can be earned even if it makes the configuration of a fiber cross section flat or incurvates it. That is, the outline configuration of a fiber cross section is made into configurations, such as an approximate circle form, an abbreviation ellipse form, and a rough convex polygon, and surface area is earned by forming much minute irregularity in a front face. Specific surface area can be enlarged without spoiling the workability and reinforcement when processing it into yarn, textile fabrics, the felt, etc. by doing in this way.

[0013] Drawing 2 is the mimetic diagram showing the example of the cross-section configuration of the titanium fiber of this invention. The outline configuration of the cross section of the titanium fiber 8 shown in this drawing 2 is an ellipse form, and has much minute irregularity on the front face. In addition, it can ask for specific surface area also including irregularity with a minute front face by measuring gas adsorption surface area with a BET adsorption method.

[0014] As construction material of the titanium fiber of this invention, pure titanium as shown, for example in a table 1, alpha alloy, an alpha-beta alloy, and beta alloy can be used. In addition, a table 1 shows the standard annealing heat treatment conditions over titanium, and is an extract from "the processing technique of titanium" for Japanese titanium associations.

[0015]

[A table 1]

[0016] Next, it explains, referring to drawing 1 about the gestalt of implementation of the manufacture approach of the titanium fiber which is the 2nd of this invention. The manufacture approach of the titanium fiber of this invention is a thing about the approach of manufacturing titanium fiber by the focusing core wire method. The process which bundles several coat many strands 4 with which the enveloping layer 2 was formed in the perimeter of the metal strand 1, and is made with the bonnet compound wire rod 6 by the sheathing material 5, Cold drawing processing and the above mentioned annealing heat treatment are repeated and performed to this compound wire rod 6, the compound strand 7, the process to make, and the part equivalent to the enveloping layer 2 and sheathing material 5 in this compound strand 7 are removed, and it has the following description in the manufacture approach of a metal fiber including the metal fiber bundle 8 and the \*\*\*\* process to make.

[0017] (b) The metal strand 1 should be titanium fiber or a titanium-alloy wire rod. For example, pure titanium as shown in the aforementioned table 1, alpha alloy, an alpha-beta alloy, and beta alloy are used.

[0018] (b) The enveloping layer 2 of the coat strand 4 and the sheathing material 5 of the compound wire rod 6 should be the mild steel containing 0.12 or less % of the weight of carbon. If it says especially, the construction material of the enveloping layer 2 of the coat strand 4 will be important, it will be desirable to make the sheathing material 5 of the compound wire rod 6 into the same construction material as the enveloping layer 2 of the coat strand 4, and this will be for making easy setting out of the annealing heat treatment conditions mentioned later.

[0019] One of the reasons which specifies especially the construction material of this enveloping layer 2 is for forming much irregularity in the front face of the titanium fiber 8 manufactured, and earning specific surface area. That is, mild steel is a polycrystal ingredient which has the crystal

structure of a body-centered cubic lattice, and each crystal grain has the strong anisotropy to deformation. For this reason, if wire drawing of the compound wire rod 6 which bundled several coat many strands 4 which made mild steel the enveloping layer 2 and made titanium the core material 1, and was covered by the sheathing material 5 is carried out, as typically shown in drawing 4, each crystal grain of the mild steel which forms the enveloping layer 2 will carry out bow deformation in the cross section, and much irregularity will be formed in the front face of the titanium core material 1. For this reason, the specific surface area of the titanium fiber 8 removed and obtained will increase the part equivalent to an enveloping layer 2 and a sheathing material 5.

[0020] Since the crystal grain of an enveloping layer 2 deforms almost isotropic in wire drawing on the other hand when crystal ingredients, such as copper with the crystal structure of a body-centered cubic lattice, are made into an enveloping layer 2, when mild steel is made into an enveloping layer 2, the irregularity of a like is not obtained, but becomes the cross section as typically shown in drawing 3, and cannot be said to be a desirable gestalt.

[0021] In the manufacture approach of the titanium fiber of this invention, since specifying 0.25 or less % of the weight of the mild steel containing 0.12 or less % of the weight of carbon preferably as construction material of an enveloping layer 2 also especially in the polycrystal ingredient which has the crystal structure of a body-centered cubic lattice has low ingredient cost, workability is good and is because formation of the coat strand 4 is easy. If it is in carbon steel with which especially a carbon content exceeds 0.25 % of the weight, it is large, and it is in the middle of processing, and it is not [ whenever / by wire drawing / hardening / needs to increase the count of heat treatment, and ] desirable. Moreover, in heat treatment which made the highest attainment temperature 580-650 degrees C, it becomes difficult to fully recover wire drawing nature. 0.25 or less % of the weight, with the above-mentioned technical problem being solvable, especially the thing that contains 0.12 or less % of the weight of carbon if it says is excellent in bending or weldability, and it becomes easy to form [ of an enveloping layer ] it.

[0022] Moreover, formation of the coat strand 4 can be easily carried out by covering the perimeter of the titanium wire rod 1 with sheet metal, such as SPCC and SPCE. Moreover, in order to make deep the depth of the surface irregularity of the titanium fiber 8 manufactured and to obtain titanium fiber with a more large specific surface area, it is desirable to thicken thickness of the mild steel enveloping layer 2 relatively to the diameter of the coat strand 4, but if it is made not much thick too much, it will become easy to produce the problem that the time amount which the \*\*\*\* process which removes the part equivalent to an enveloping layer 2 and a sheathing material 5 takes becomes long. Then, the range where the thickness of an enveloping layer 2 is desirable is 5% to 20% of the diameter of the coat strand 4, and the still more desirable range is 8% to 15%.

[0023] And although whenever [ bow / of each crystal grain of mild steel ] becomes large and titanium fiber with a large specific surface area is obtained so that the amount of cold drawing processings given to the compound strand material 6 which bundled several coat many strands 4 and was covered by the sheathing material 5 is enlarged, it becomes easy to produce the problem that the time amount which a \*\*\*\* process takes too becomes long. Then, total amount of processings  $\epsilon_T$  of the cold drawing given to a compound wire rod (it is desirable to set  $\epsilon_T = 2 \times \ln(DS/DF)$  (formula 3) to 5.5 to 7.5.) It is DS here. The diameter of the compound wire rod 6 before performing cold drawing processing, and DF It is the diameter of the compound strand 7.  $\epsilon_T$  Since whenever [ bow / of the crystal grain of mild steel ] is small

when it is 5.5 or less, the irregularity of titanium fiber is small, and specific surface area becomes so large and is not desirable, either. Moreover, it becomes [ in the case of 7.5 or more the surface irregularity of titanium fiber becomes intense, and / that the irregularity of the metal fiber adjoined in a compound wire rod becomes entangled mechanically, and they dissociate at a \*\*\*\* process ] difficult and is not desirable.

[0024] (c) Make into 580 to 650 degrees C the highest attainment temperature of the compound wire rod in annealing heat treatment performed to the compound wire rod 6. This temperature requirement is a temperature requirement where this invention persons set up an experiment and examination in piles about the annealing heat treatment conditions of giving many coat strands 4 with which the mild steel enveloping layer 2 was formed in the perimeter of the titanium strand 1 to the compound wire rod 6 further covered by the mild steel sheathing material 5 in \*\*\*\*.

[0025] The standard heat treatment conditions over the titanium which does not have a mild steel coat were understood that it is required to take into consideration the diffusion phenomenon in the interface of titanium and mild steel with the softening degree of the compound wire rod 6 in annealing heat treatment of the compound wire rod 6 containing the coat strand 4 with which the mild steel enveloping layer 2 was formed in the perimeter of the titanium strand 1 the place which is a thing as shown in the above mentioned table 1. Namely, if the highest attainment temperature exceeds 650 degrees C, since the alloy layer formed of diffusion of the interface of titanium and mild steel will progress, When an enveloping layer 2 tends to be removed and it is going to obtain titanium fiber 8, even if clearance of an enveloping layer 2 becomes difficult and titanium fiber 8 is obtained When the highest attainment temperature is less than 580 degrees C, on the other hand in core wire processing between the colds which the softening degree of the compound wire rod 6 serves as imperfection, and continues, it becomes easy only for a part of titanium fiber in the compound strand 7 to be obtained, but for the yield to fall remarkably, and to produce an open circuit remarkably.

[0026] Thus, although it is required to make into 580 to 650 degrees C the highest attainment temperature in annealing heat treatment performed to the compound wire rod 6 at least in the manufacture approach of the titanium fiber of this invention When the process which forms the coat strand 4 before forming the compound wire rod 6 includes annealing heat treatment to the covered-wire material 3 which formed the mild steel enveloping layer 2 in the titanium core material 1, it is desirable to also make into the range of 580 to 650 degrees C the highest attainment temperature in annealing heat treatment performed to the covered-wire material 3. In addition, since titanium [ activity / front face ] is covered with mild steel when performing annealing heat treatment to the compound wire rod 6 or the covered-wire material 4, it can operate also by the furnace atmosphere applied to steel-wire material using a gas fired furnace, an electric furnace, etc.

[0027] In addition, in the manufacture approach of the titanium fiber of this invention, especially wire drawing performed to the compound wire rod 6 is considered as cold drawing processing for an alloy layer tending to develop into the interface of titanium and mild steel, in order to ease the anisotropy over processing and for the effectiveness of surface irregularity formation for titanium fiber to fall, if hot working in an elevated temperature is applied. As the approach of cold drawing processing, the dry wire drawing method by the hole dice, a wet drawing method, or a roller die is applicable. Moreover, since it is covered with mild steel, wire drawing of the front face of the compound wire rod 6 or the covered-wire material 4 can be carried out using the lubricant for wire drawing of steel wire.

## EXAMPLE

[Example] It explains to a detail further with the desirable example of this invention. The description of the titanium fiber 8 manufactured on five kinds of manufacture conditions shown in a table 2 through the yield and \*\*\*\* process of an attempt, the stability of a process, and the titanium fiber in a \*\*\*\* process in manufacture of the compound strand 7 which included much titanium fiber was compared.

[0029]

[A table 2]

[0030] In a table 2, an example 1 and an example 2 are examples according to the suitable conditions of the manufacture approach of the titanium fiber of this invention, and have set up more thickly than an example 1 the enveloping layer thickness of the covered-wire material 3 in an example 2. In addition, in order to make equivalent to an example 1 the circle equivalent diameter of the titanium fiber 8 manufactured in an example 2, it is the example which has set up the diameter of the final compound strand 7 thickly a little rather than the example 1.

[0031] The example 1 of a comparison is an example which set up lower than the range according to this invention the highest attainment temperature in annealing heat treatment of the compound wire rod 6. Moreover, the example 2 of a comparison is an example which set more highly than the range according to this invention the highest attainment temperature in annealing heat treatment of the compound wire rod 6 as reverse in the example 1 of a comparison. The example 3 of a comparison is an example which used copper as an enveloping layer 2. In addition, since it is hard to diffuse copper in titanium compared with iron, it gave priority to the softening degree of the titanium by heat treatment in the example 3 of a comparison, and has set up the highest attainment temperature more highly than other examples.

[0032] In formation of an enveloping layer 2, the pure titanium core material 1 was inserted in the interior, forming a welded tube with a diameter of about 6mm with the strip made into an enveloping layer 2, wire drawing was carried out to the diameter of 4.3mm, and the wall and core material front face of tubing were stuck. The strip made into an enveloping layer 2 used the copper strip for the example 1 and the example 2, and the list in the example 3 of a comparison using the strip of SPCC in the example 1 of a comparison, and the example 2 of a comparison. Moreover, in formation of the compound wire rod 6, the bundle of the coat strand 4 was inserted in the interior, forming a welded tube with a diameter of about 6mm with the strip of SPCC, and wire drawing was carried out to the diameter of 4.3mm, and it screwed up.

[0033] Heat treatment was performed continuously through the wire rod into the electric furnace set as predetermined temperature, except having made the heat treatment ambient atmosphere of the covered-wire material 3 in the example 3 of a comparison into the inert atmosphere, it was heat-treated by the weak oxidizing atmosphere, and the wire drawing which continues after making a front face into clarification by acid washing and rinsing was presented with it. Wire drawing applied the cold drawing by dry type and wet, and performed it using the lubricant for steel wire.

[0034] the result of having tried manufacture of the compound strand 7 which included much titanium fiber 8 on five kinds of manufacture conditions shown in a table 2 -- the heat treatment temperature of the compound wire rod 6 -- the likeness of excess -- in the example 1 of a comparison set up low, although the compound strand 7 of the diameter which open circuits

occur frequently in the last wet drawing process, and is made into the object was not obtained. The compound strand 7 of the diameter made into the object in other examples was able to be obtained.

[0035] In a concrete production process, if a carbon content and workability are described, and the charge of carbon content uses the carbon steel which is about 0.55 % of the weight, for example, the part welded at the time of welded tube formation will break at the time of wire drawing, and will not bear processing on the way. Moreover,  $\epsilon$  If attached, if the example 2 was in the example of 6.14 and others, it was 6.35, and all could carry out wire drawing and \*\*\*\* processing satisfactory, and the titanium fiber of the specific surface area made into the object was obtained.

[0036] Then, \*\*\*\* processing was performed to the compound strand 7 manufactured at four kinds of processes except the example 1 of a comparison, and the description of the titanium fiber 8 obtained with the yield of an attempt and a \*\*\*\* process in manufacture of the titanium fiber bundle 8 was compared. The result is shown in a table 3. In addition, \*\*\*\* processing was performed by electrolyzing selectively the part equivalent to the sheathing material 5 and enveloping layer 2 in the compound strand 7 in the electrolytic solution containing a sulfuric acid.

[0037] The yield of a \*\*\*\* process is the rate separated as titanium fiber 8 among the titanium wire rods which the longest also made 1 hour time amount of electrolysis at a \*\*\*\* process, and were laid underground into the compound strand 7. Moreover, measurement of the specific surface area of titanium fiber 8 measured the nitrogen amount of adsorption with the BET adsorption method, and it was converted into the surface area per unit quantity, and it evaluated it.

[0038]

[A table 3]

[0039] As shown in a table 3, even if it covered the compound strand 7 of the example 2 of a comparison which set up highly the heat treatment temperature of the compound wire rod 6 too much over electrolysis time amount in the \*\*\*\* process for 1 hour, it could not be \*\*\*\*(ed) thoroughly, but its yield was remarkably low. On the other hand, the compound wire rod 7 of the example 1 and example 2 according to the manufacture approach of this invention was able to be thoroughly \*\*\*\*(ed) by electrolysis of less than 1 hour. The cross section of the obtained titanium fiber 8 is a configuration as shown in drawing 2 , and much irregularity was formed in the front face, and specific surface area was so large that it satisfied the formula 1, and was able to adsorb a lot of nitrogen gas per unit weight. Moreover, in the example 2 set up more thickly than the thickness of an enveloping layer 2, titanium fiber with a larger specific surface area than an example 1 was able to be obtained.

[0040] Moreover, although the compound strand 7 of the example 3 of a comparison which used construction material of an enveloping layer as copper was able to be \*\*\*\*(ed) thoroughly, its front face of the obtained titanium fiber 8 was smoother than the front face of the titanium fiber chosen from the compound wire rod 7 of an example 1 and an example 2. And the specific surface area was smaller than the titanium fiber obtained from the compound wire rod of an example 1 and an example 2, and was not what satisfies a formula

## DESCRIPTION OF DRAWINGS

### [Brief Description of the Drawings]

[Drawing 1] Drawing 1 is process drawing of the manufacture approach of the titanium fiber of this invention.

[Drawing 2] Drawing 2 is the mimetic diagram showing the example of the cross section of the titanium fiber of this invention.

[Drawing 3] Drawing 3 is a mimetic diagram which is obtained by the manufacture approach of the conventional metal fiber and in which showing the example of the cross section of titanium fiber.

[Drawing 4] Drawing 4 is the mimetic diagram in the cross section of the compound strand according to the manufacture approach of the metal fiber of this invention showing the condition near the interface of mild steel and titanium.

### [Description of Notations]

- 1 .... Core material,
- 2 .... Enveloping layer,
- 3 .... Coat wire rod,
- 4 .... Coat strand,
- 5 .... Sheathing material,
- 6 .... Compound wire rod,
- 7 .... Compound strand,
- 8 .... Metal fiber.

(DRAWINGS IN JAPANESE PUBLICATION ESSENTIALLY THE SAME AS IN US PATENT 6,325,875)